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# NEW METHOD OF EXPERIMENTAL WORK AT HIGH TEMPERATURES

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/Digest/

The experimenter working at temperatures above 2,000° C does not have at his disposal an inert crucible; only in rare cases is it possible to choose a crucible material which does not react. Occasionally it is possible to select a crucible material which as such forms one of the solid phases of the reaction under investigation; in such cases one speaks of a so-called equilibrium crucible. However, this limits the investigation to the range of equilibrium of only one of the system's solid components.

Under the circumstances, methods which permit working without a crucible are of considerable interest. One such method may be based on the precise condition which precludes the use of a crucible, namely, the high velocity of processes taking place at elevated temperatures. At sufficiently high temperatures, the velocity of most processes (including heat transfer by radiation) increases to such an extent that it becomes possible to complete many processes during the time of the free fall through a furnace of a small particle of the investigated substance.

The numerical values of the velocities of most processes at high temperatures are not known. For this reason, one may compute only the processes of simple temperature equalization and melting (or, generally speaking, any isothermic phase transformations the velocity of which depends only on the rate of heat supply). The Stefan-Boltzmann law gives the following expression for the time $Z_1$ (in seconds) necessary for equalization of the temperature between a heated, enclosed sheath (a furnace) and a small spherical body placed in it which is so small that the temperature differences between its various points may be neglected:



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$$Z \cong \frac{D\gamma c}{\alpha} \; 6.05 \times 10^{10} \left[0.785 + 1.15 \lg \frac{2T_1 (T_1 - T_2)}{\Delta T \; (T_1 + T_2)} - \arctan \frac{T_2}{T_1}\right] \qquad (1)$$

where D is the diameter in cm, $\gamma$ the density (in $g.cm^{-3}$), c the specific heat capacity $\frac{cal}{g.^oC}$, $\alpha$ the coefficient of radiation, $T_2$ the initial temperature (in $^oK$) of the body, $T_1$ the temperature (in $^oK$) of the uniformly heated, enclosed sheath (i.e., the furnace), and $\Delta T$ the allowed temperature difference between the body which is being heated and the furnace.

According to this expression, the time necessary for temperature equalization between the furnace and the body being heated under heat transfer by radiation only diminishes rapidly with increased temperatures of the furnace.

Thus, for practical application of the proposed method, the furnace should become shorter with increasing experimental temperatures.

The time $Z_2$ (in seconds) which is necessary for completely melting a small spherical body (or, in general, for completing any isothermic phase transformation the velocity of which depends only on the rate of heat supply) is given by the expression:

$$Z_2 = \frac{D\gamma L}{\alpha} \cdot \frac{1.21 \times 10^{11}}{T_1^4 - T_n^4}$$

where L is the heat of phase transformation (in $cal.g^{-1}$) and $T_n$ the temperature of phase transformation (in $^oK$).

Or, if $T_1 - T = \Delta T$ (the allowable difference between the temperature of the furnace and the temperature of phase transformation) is of small magnitude in comparison with $T_1$, the following relationship will hold true:

$$Z_2 \cong \frac{D\gamma L}{\alpha} \cdot \frac{0.303 \times 10^{11}}{T^3 \cdot \Delta T} \qquad (2)$$

This expression gives the time of melting at the allowable temperature difference between the furnace and the substance under investigation. Or, in other words, the time during which a sample previously brought to its melting temperature melts in a furnace which is also at the melting point temperature of the sample (within the limits of deviation $\Delta T$) is given by the expression (2).

Expressions (1) and (2) permit calculation of the time during which a sample having the temperature $T_2$ melts after having been introduced into a furnace the temperature of which exceeds by $\Delta T$ the temperature of the sample.

From the practical standpoint, the experimenter may use a furnace having a heated tube that is 100 cm long, which corresponds to a 0.4-second duration of the free fall. If a furnace of this length is used and the measurement is carried out on grains of a diameter no smaller than 0.03 cm, the proposed method can be applied, as a calculation on the basis of various substances shows, at temperatures in excess of 2300° C, provided that one is satisfied with a precision of temperature equalization amounting to $\Delta T = \pm 30^o$ C. Under those conditions, the possible temperature differential between the center of the grain and its surface does not exceed a few degrees and consequently may be neglected.





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For checking experimentally the suitability of the proposed method, I determined the melting points of several high melting substances on which reliable data could be found in the literature.

The construction and operation of the equipment used in this series of experiments are described below.

A graphite tube of the following dimensions was used as the furnace: inner diameter 1.7 cm, outer diameter 2.4 cm, length 80 cm. Heating was effected by passing an electric current of 200-500 a and 20-30 v directly through the tube. The tube was placed in a cooling jacket, and soot [carbon black] was used for heat insulation. A furnace of this type is placed vertically. At the bottom, the heating tube of the furnace is connected with a glass tube in such a manner that both are disposed coaxially. The connection of the graphite tube with the glass tube, as well as the electrical contacts, is cooled with water. During the operation, nitrogen is passed through the furnace. The upper end of the heating tube is equipped with an appliance by means of which grains of the substance under investigation are dropped through the heating tube. The grains, upon dropping through the heating tube of the furnace, pass through the glass tube (in which they are cooled), and drop into a receptacle placed below.

For temperature measurement, the heating tube is provided with a hole having a diameter of 0.8 cm, situated in the middle of the tube. A small horizontal carbon tube passes through this hole after penetrating through the furnace jacket. This tube serves for measuring the temperature of the inner surface of the heating tube by means of an optical pyrometer. During the temperature determination, nitrogen is blown through the horizontal tube. The end of the tube facing the potentiometer is equipped with a plane-parallel glass port. A disappearing thread pyrometer which had been graduated by means of a standard wolfram filament bulb and a rotating sector (serving as a grey screen) for reducing the radiation of the furnace were used. The precision of the temperature measurement was $\pm$ 30° C.

Special measurements by means of a movable black body model showed that the temperature was sufficiently constant along a 65-cm stretch of the axis of the heated tube in the tube's middle portion.

The substances to be investigated were formed into tablets and calcined (Mo - W mixtures in an $H_2$ atmosphere). The tablets were powdered and the powder was sifted in order to obtain grains having dimensions of 0.2-0.5 mm. The grains were dropped through the furnace at various temperatures. The lowest furnace temperature at which the grains of a substance were melted into spheres was considered to be the melting point of the substance in question.

One may note that substances like CaO and BeO, which usually can not be melted in a furnace equipped with a carbon tube without undergoing reduction by the carbon (this being the reason why all attempts to determine their melting point in a carbon furnace resulted in values which were too low), melted into perfectly white spheres when the above-described procedure was applied. The CaO spheres obtained in this manner dissolved in water without evolution of gas, showing that no $CaC_2$ was present. Some of the spheres contained adhering graphite due to the fact that they hit the walls of the tube. These spheres could be eliminated by visual selection.

Table 1 compares some values obtained by the new method with the most probable values obtained by older methods.

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Up to the present, the newly proposed method has been used by me for determining the melting points of individual chemical substances and also for determining the melting temperatures in solutions in which the solidus curve lies close to the liquidus curve, as for instance in the system Mo - W.

It is possible that the velocity of still other processes is great enough to use the method for the study of the equilibria in question at temperatures exceeding 2,300-2,400° C.

Table 1. Comparison of Melting Points Obtained by the New Method With Values Obtained by Older Methods

| | CaO | BeO | Mo | 70% Mo 30% W | 50% Mo 50% W |
|---|---|---|---|---|---|
| Determined melting points, in °C | 2620 | 2570 | 2450 | 2700 | 2820 |
| Melting points according to published data | 2585* | 2570* | 2420** | 2680** | 2850** |

* O. Ruff, Z. Angew. Chemie, Vol XLVI, 1, 1933.

** M. Hansen, Structure of Binary Alloys (in Russian), Vol II, 1941, 872.

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